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Light scattering methods to test inorganic PCMs for application in buildings

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Abstract. Thermal performance and stability over time are key parameters for the characterization and application of PCMs in the building sector. Generally, inorganic PCMs are dispersions of hydrated salts and additives in water that counteract phase segregation phenomena and subcooling. Traditional methods or in “house” methods can be used for evaluating thermal properties, while stability can be estimated over time by using optical techniques. By considering this double approach, in this work thermal and structural analyses of Glauber salt based composite PCMs are conducted by means of non-conventional equipment: T-history method (thermal analysis) and Turbiscan (stability analysis). Three samples with the same composition (Glauber salt with additives) were prepared by using different sonication times and their thermal performances were compared by testing both the thermal cycling and the thermal properties. The stability of the mixtures was verified by the identification of destabilization phenomena, the evaluation of the migration velocities of particles and the estimation of variation of particle size.

1. Introduction

Dispersions based on Glauber salt can be very interesting for applications in the building sector, thanks to the high latent heat of fusion and the temperature at which this transition occurs. On the other hand, the applicability of such kinds of dispersions can be limited by diverse problems.

First of all, the initial particles diameters and destabilization phenomena could compromise the reproducibility of the thermal properties over the time.

In fact, dispersions are unstable systems from a thermodynamic point of view, characterized by a gradual destabilization following different kinetics and mechanisms. In a very slow process, these phenomena are not easily detected by the operator’s naked eye but they cause phase separation after some time and decreasing thermal performances in PCMs [1, 2].

It was also demonstrated that the modality of preparation, and in particular the sequence of the reagents addition, influences the initial characteristics of the dispersion [3].

Moreover, sub-cooling and phase segregation problems occur and require the addition of appropriate additives [4].

For example, tetraborate decahydrate (borax) is often used as nucleating agent, while fumed silica, bentonite, or polymeric gels are used as thickeners [5].



In this work, borax was chosen as a nucleating agent because of the positive results obtained in different studies [6], while bentonite was preferred to other thickeners due to the good price-performance ratio. On the other hand, the use of bentonite needs attention due to the strong bonding and van der Waals forces, and its dispersibility requires intense mixing forces to break the agglomerates uniformly.

Generally, dispersions are also affected by particle size variation. In particular, the authors underlined the presence of flocculation in previous investigations [4].

For these reasons, in this investigation sonication was applied to facilitate the disaggregation of agglomerates and sodium hexametaphosphate was used in order to reduce the extent of flocculation.

Furthermore, previously tested sample preparation procedures were adopted [7] and the aim of the study was focused on the influence of sonication time on the thermal properties and texture of the analyzed dispersions.

The determination of the thermal properties can be obtained by means of traditional methods as DSC or in “house” methods as T-history. T-history seems to be more suitable compared to DSC, due to the greater and more significant sampling [8]. Such a property is suitable when inhomogeneous materials have to be investigated. Several cycles of cooling and heating help to test thermal stability [9]. This procedure allows the detection of performance degradation during the time but is not able to detect and classify eventual destabilization processes.

Recently, some studies joined more detailed investigations on PCM structure by using the scanning electronic microscope (SEM) and Fourier transform infrared spectrophotometer (FTIR) [10].

By considering this double approach, in this work thermal and structural analyses of inorganic PCMs Glauber salts/additives are conducted.

The samples obtained by different sonication times are tested from the thermal point of view. In particular, solidification enthalpy (ΔH) and the solidification range are evaluated by the T-history method.

The evolution in time of the mixture, in terms of destabilization phenomena, is followed by means of Turbiscan. It is an instrument commonly used in the pharmaceutical and cosmetic sector since it allows identification of slow and not visible phenomena that precede dispersion destabilization [11]. Its operation is based on multiple light scattering associated with vertical scanning of the sample, which allows a rapid evaluation of both the occurrence and the typology of destabilization phenomena. The particular advantage is the ability to monitor, without dilutions and in a continuous way, the PCM behavior in operating conditions, working on volumes of samples more significant than those used in the methods based on microscopy techniques. In this investigation, Turbiscan was used for the evaluation of the migration velocities of particles and the estimation of variation of particle size.

2. Materials and methods

2.1. Reagents

The samples were obtained as mixtures of the following reagents: anhydrous sodium sulfate, sodium tetraborate (borax) decahydrate, bentonite and sodium hexametaphosphate. They were purchased from Sulquisa (Bilbao, Spain), Carlo Erba (Milan, Italy), and Sigma-Aldrich (Milan, Italy), respectively.

2.2. Instruments

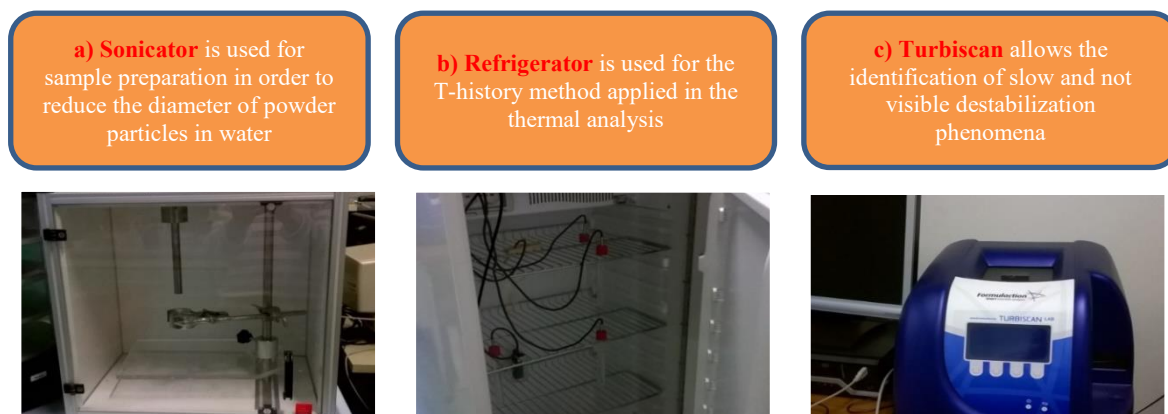
The experimental set-up consists of three main components: sonicator, refrigerator, and Turbiscan. They are used for preparation, thermal cycling, and optical analysis, respectively. The description of the technical characteristics of these instruments is presented below.

The sonicator is provided by Hielscher (Teltow, Germany). UP200S compact ultrasonic homogenizer features include sturdiness, high degree of efficiency, and automatic determination of the ideal performance control by way of the amplitude or the pulse duration. The characteristics of the sonotrode are summarized in table 1.

Table 1. Characteristics of the sonotrode used for samples preparation.

Probe for sonication				
Type	Max immersion	Diameter TIP	Max amplitude	Acoustic Power Density
S14 - TIP 14	90 mm	14 mm	80 μm	105 W/cm ²
Amplitude	Cycles			
100%	0.8			

Thermal cycling is carried out by the refrigerator FOC 2151 (Usmate Velate (Monza Brianza), Italy) that is a high-efficiency (energy label A+) refrigerated incubator provided with an innovative Auto-Tuning thermoregulation system, and forced air circulation in order to offer excellent stability and homogeneity of the internal temperature. The stability analysis was developed by Turbiscan® LAB Thermo (Formulation), purchased from Alfatest Rome. It is based on multiple light scattering [12] and offers new performances to characterize concentrated dispersions without dilution. Turbiscan operates on emulsions, suspensions and foams with a concentration of up to 95% v/v, over a wide range of size (10 nm to 1 mm). It has been already used to study suspension stability in other fields than PCMs characterization. With this instrument, it is possible to identify phenomena of slow suspended particles migration - causing sedimentation or creaming – and aggregation leading to coalescence and flocculation. Moreover, it allows evaluation of the kinetic destabilization in order to compare different dispersions. The schema and equipment of the experimental setup are illustrated in figure 1.

**Figure 1.** Components of the experimental setup. a) sonicator, b) refrigerator, c) Turbiscan.

2.3. Sample preparation

The samples were prepared with the same composition, as summarised in table 2.

Table 2. Compositional weight of the tested mixtures.

Water (g)	Bentonite (g)	Sodium Sulphate (g)	Borax (g)	Sodium hexametaphosphate (g)
60	5	31	4	1

Sodium hexametaphosphate was added to limit flocculation [13] while borax was used in order to reduce subcooling and bentonite was used to improve sample stability. The first step of preparation was subdivided into three steps of 10 min sonication each; in each time, one third of the total quantity of the salts was added. Three sample were prepared following this procedure and, after bentonite

addition, were submitted to sonication for different times: Sample 1 (10 min sonication), Sample 2 (20 min sonication), Sample 3 (30 min sonication).

2.4 Thermal analysis: T-history method

The thermal analysis by T-history method was performed in the temperature range of 10-40 °C. Four different cooling/heating cycles were executed, in order to verify the thermal stability of the samples [3, 7].

The results were presented with reference to the method of Marin et al. [8], reporting the enthalpy-temperature curve in the investigated range, instead of the solidification enthalpy, calculated starting from the temperature vs time data. This is more useful, because PCMs do not have a homogeneous composition and, consequently, their solidification interval can be very large. The enthalpy-temperature curve is described by equation 1.

$$\Delta h_p(T_i) = \left(\frac{m_w \cdot c_{pw}(T_i) + m_t \cdot c_{pt}(T_i)}{m_p} \right) \cdot \frac{I_i}{I_i'} \cdot \Delta T_i - \frac{m_t}{m_p} \cdot c_{pt}(T_i) \cdot \Delta T_i \quad (1)$$

where m_w , m_t and m_p are the mass of water (reference substance), test tube and PCM, respectively [kg]; c_{pw} , c_{pt} are the specific heat of water and test material [$\text{kJ kg}^{-1} \text{K}^{-1}$]; ΔT_i is the temperature range [°C] with medium value T_i , to whom corresponds the enthalpy variation $\Delta h_p(T_i)$ [kJ/kg]; I_i is the integral with respect to time of the difference in temperature between PCM and environment; I_i' is the integral with respect to time of the difference in temperature between water and environment.

The latent heat of solidification was calculated as $\Delta h_p(T_{in}) - \Delta h_p(T_f)$, where T_{in} is the initial temperature of solidification equal to the maximum value reached after the supercooling process, and T_f is the final temperature equal to the inflection point in cooling curve [14]. Specific heat capacity for liquids (c_{pl}) and solids (c_{ps}) were calculated from the slope of the two straight lines [8]. To better compare different cycles, the effect of supercooling was not included, but reported for each test.

2.5 Stability analysis: TURBISCAN-based method

The stability of the samples was characterized by means of light scattering method based on the Turbiscan (see Fig.1c), an instrument typically used in the pharmaceutical and cosmetic sector, able to detect all kind of destabilization phenomena due to particles migration (sedimentation, creaming) or granulometric variation (flocculation, coalescence) at an early stage.

The special Turbiscan tube (the test cell) is filled with the sample and the optical analysis is carried out for the whole cell length. It works on Multiple Light Scattering in transmission (*T-mode*) and backscattering mode (*BS-mode*) and both signals depend on particle sizes and concentration. If the sample is stable, no variation in time of BS and T profile occurs, otherwise each phenomenon could be related to profile variation.

To emphasize them, the Delta Back scattering mode (ΔBS) could be used. In this view mode, one of the profiles (by default the first one), is used as a “blank” corresponding to the initial dispersion state of the sample. This profile is then subtracted from all the other profiles. ΔBS trends for the different phenomena that could occur during destabilization, such as sedimentation, flocculation/coalescence, clarification and creaming, are reported in figure 2.

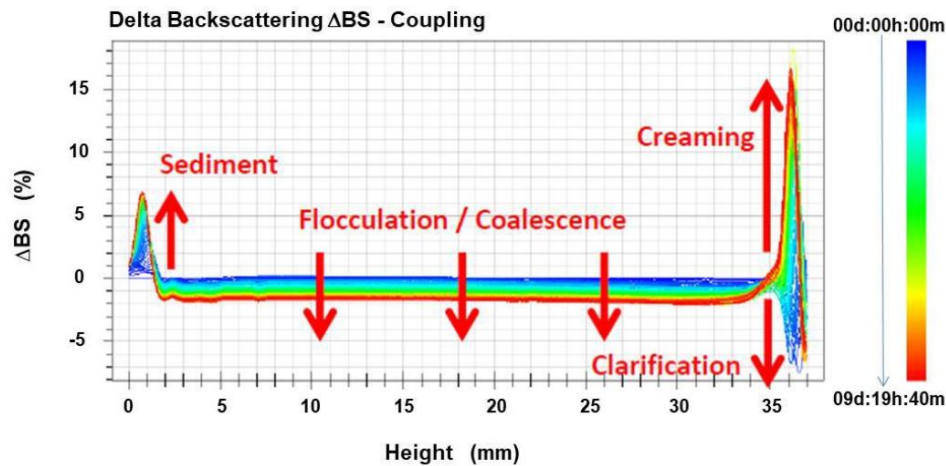


Figure 2. Destabilization phenomena in a Delta Backscattering graph [11].

Turbiscan also allows estimation of characteristic parameters related to the particular type of destabilizing phenomenon, as reported in table 3 [11].

Table 3. Parameters related to the type of destabilizing phenomenon in the analysis by Turbiscan [11].

Instability phenomena	Kinetics	Characteristic parameters
Sedimentation	<ul style="list-style-type: none"> - Clarification kinetics (phase thickness of right peak) - Sedimentation kinetics (phase thickness of left peak) - Mean value (delta BS or delta T) at the top and bottom 	<ul style="list-style-type: none"> - Migration velocity (slope of clarification kinetics) - Hydrodynamic diameter
Creaming	<ul style="list-style-type: none"> - Clarification kinetics (phase thickness of left peak) - Creaming kinetics (phase thickness of right peak) - Mean value (delta BS or delta T) at the top and bottom 	<ul style="list-style-type: none"> - Migration velocity (slope of clarification kinetics) - Hydrodynamic diameter
Particle size increase	<ul style="list-style-type: none"> - Mean value kinetics in the middle - Variation of l^* in the middle - Variation of the mean diameter in the middle (for non-absorbing, white product) 	<ul style="list-style-type: none"> - % particle size increase (slope of the mean value kinetics)
Ripening of a foam	<ul style="list-style-type: none"> - Mean value kinetics in the middle - Variation of l^* in the middle - Variation of the mean diameter in the middle (for non-absorbing, white product) - Phase thickness of the syneresis phase (transmission peak at the right) 	<ul style="list-style-type: none"> - % particle size increase (slope of the mean value kinetics)

If particle migration occurs at the end of the cell, it is possible to calculate the speed of the phenomenon expressed as the slope of the curve which is obtained by recording the variation in the width of the peak in time. Concerning the phenomena of variation of the particle size, the slope of the curve which is obtained by recording the variation of the average backscattering over time allows evaluation of the percentage of increase in particles diameter. The sample was poured into the TURBISCAN glass cell and thermally stabilized for five minutes prior to analysis. Each sample was

analyzed every 5 minutes for 24 hours at 40°C (the maximum investigated temperature), to highlight destabilization due to lower viscosity.

3. Experimental results

3.1 Thermal analysis

The thermal properties of the three samples were analyzed by the T-history method. Four cooling and heating cycles were completed for each sample. The results obtained for sample 1 are reported in figure 3: in this figure, the temperature profiles of the sample, of the reference substance (water) and of the heating-cooling chamber where the T-history method is applied (ambient) are reported as a function of time. Similar behaviours have been observed for the other samples.

The results were then processed in order to calculate the fundamental thermal properties: solidification temperature range, solidification enthalpy, specific heat both in the liquid and solid phases.

First of all, from the cooling curves we have calculated the enthalpy-temperature curves for each sample, in order to verify their thermal stability. In such an analysis, the effect of supercooling has been neglected for all the samples, because it was lower than 5 °C at any time. The results are shown in figure 4.

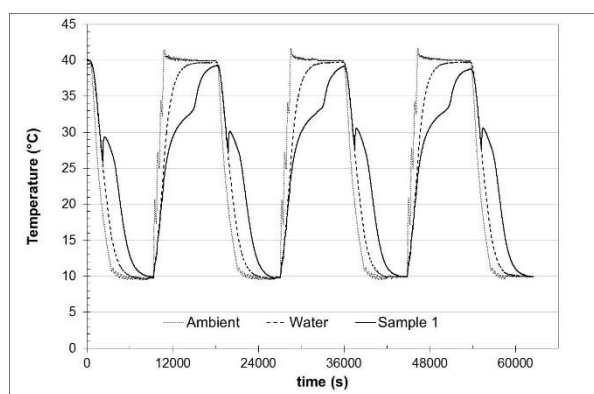


Figure 3. Thermal cycles of sample 1 obtained in the range of temperature 10–40 °C, with the T-history method, in comparison with thermal cycle of water (reference) and ambient (heating-cooling chamber FOC 215).

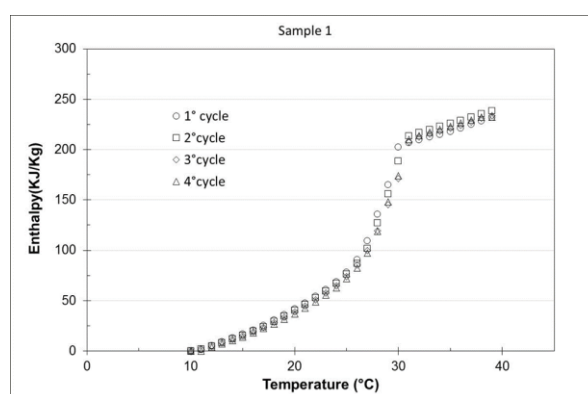


Figure 4. Enthalpy-Temperature curves for sample 1 in the four cycles of cooling-heating during the T-history method.

No significant differences were observed during the four cycles and the same behaviour was found for samples 2 and 3. This result permits the authors to conclude that all the samples are characterized by a good thermal stability in terms of heat storage. Consequently, the comparison between their performances is reported only for the first cycle and shown in figure 5.

The comparison between the three curves permits the highlighting of the dependency of the melting/solidification enthalpy value (ΔH) on the last step of sonication of the sample and in particular on the sonication time.

Different values of solidification temperature could be measured for the samples, though it should be possible to identify a common solidification temperature range for the three samples, where their phase change is observed. Such a range has been selected equal to 25–35°C, as evidenced in figure 5.

Furthermore, ΔH was calculated for each sample and the results were correlated as a function of the sonication time, as reported in figure 6. Solidification enthalpy increases with the sonication time: moreover, such an increment is more significant between 10 and 20 minutes of sonication (an increase

of about 3.5 kJ/kg min) than from 20 to 40 min (an increase estimated in 0.6 kJ/ kg min). Some considerations could be done in order to explain such behaviour. It is possible to assume that the most effective disintegration of the obtained crystals of sodium sulfate could be reached by increasing sonication time. In fact, initially the crystals may be present in the form of agglomerates whose existence could constitute a limit to the formation of their decahydrate form (Glauber's salt) which offers the best thermal performance. An effective sonication could disaggregate such an agglomerate and an effective Glauber's salt could be obtained. However, it seems possible to find a limit beyond which no significant improvement could be obtained in terms of material texture and, consequently, in terms of thermal properties.

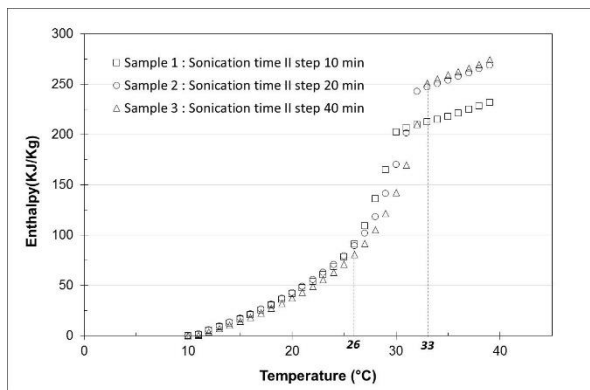


Figure 5. Comparison between the three samples in terms of enthalpy-temperature behaviour during the first cycle of cooling.

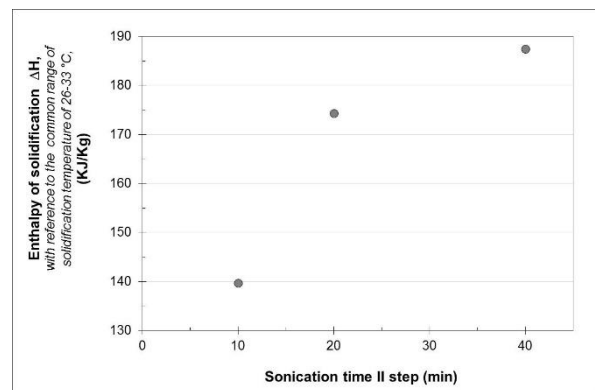


Figure 6. Solidification enthalpy ΔH calculated in the common solidification temperature range of 26-33 °C for the three samples, as function of sonication time.

Further considerations could be also done with reference to the supercooling effect observed for all the samples in relation to the sonication time. The temperatures of the initial solidification and the supercooling are reported in table 4.

Table 4. Temperature of initial solidification and supercooling degree at variation of sonication time.

	Temperature of initial solidification	Supercooling degree
Sample 1	30.03 °C	3.03 °C
Sample 2	31.87 °C	3.72°C
Sample 3	32.22 °C	4.48°C

The different texture, in terms of higher concentration of effective Glauber's salt, achieved with longer sonication times, confirm that the increase of initial solidification temperatures corresponds to an increase of supercooling degree. The disintegration of crystals and, consequently, the virtual increase of Glauber's salt concentration (in terms of effective availability for the process) could reduce the virtual ratio from borax (used in order to reduce the supercooling) and Glauber's salt, losing some amount of latent heat. The reduction of such a ratio could be the cause of the increase in supercooling.

From an analysis of figure 6 and table 3, it seems that it is possible to find an optimal or limit value of sonication time, where ΔH could be improved without a significant increase of supercooling.

3.2 Destabilization phenomena characterization

The characterization of the texture of the sample has been carried out, as already stated, by means of multiple light scattering analysis, with the Turbiscan instrument. All the samples were opaque, and as

a consequence, during the analysis in Turbiscan, the characterization of destabilization phenomena was done taking into account the ΔBS profiles along the measurement cell.

In figures 7.a, 7.b and 7.c the ΔBS behaviour along the height of the cell is presented for samples 1, 2 and 3, respectively.

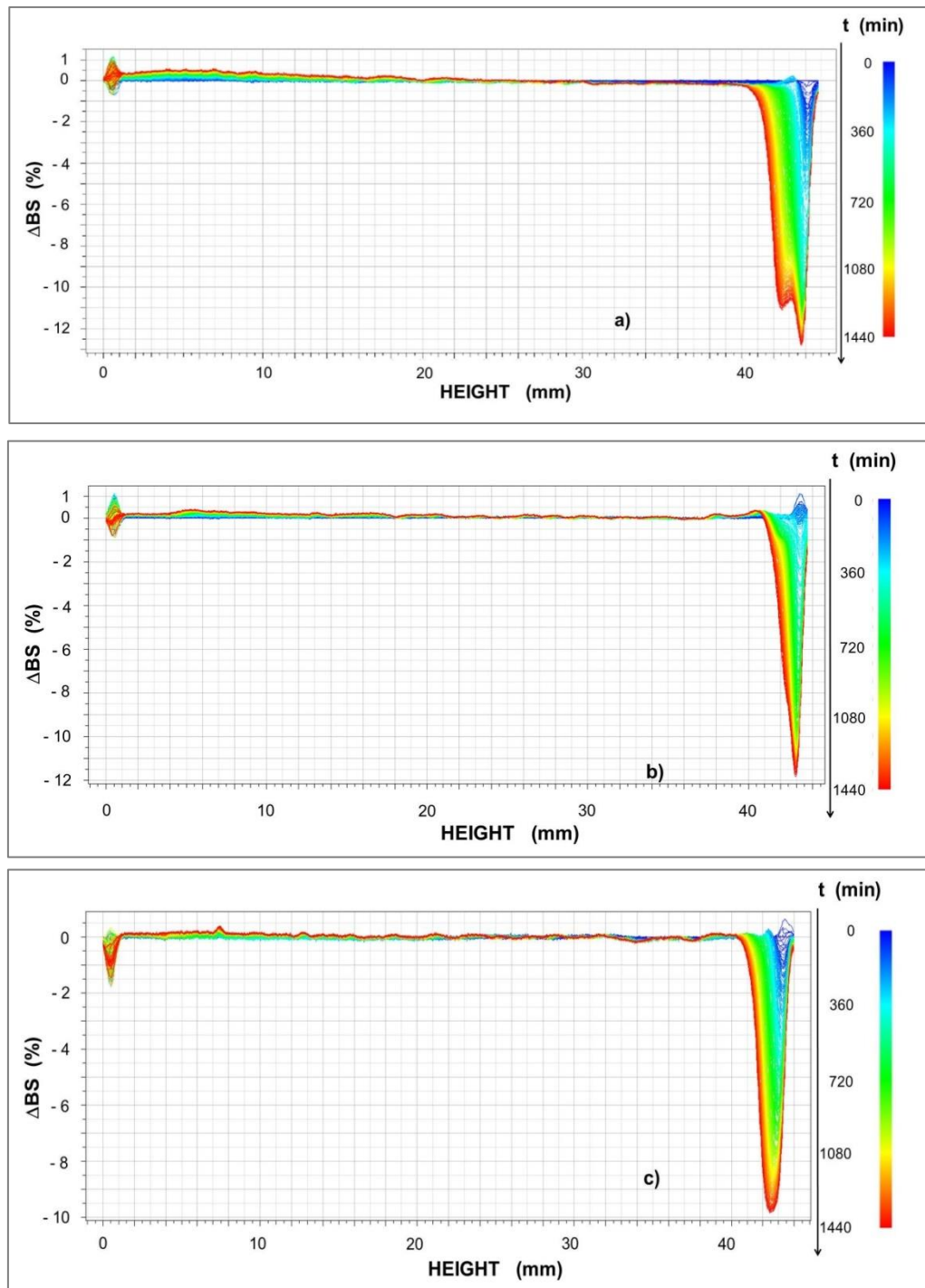


Figure 7. Delta backscattering ΔBS behaviour along the cell during Turbiscan analysis for the samples: a) sample 1 (sonication time of II step of 10 min); b) sample 2 (sonication time of II step of 20 min); c) sample 3 (sonication time of II step of 40 min).

The trends obtained for the three samples are qualitatively similar and it is possible to assume that they are characterized by the same destabilizing phenomena. As consequence, the quantitative analysis will be shown just for the first sample, but all the obtained results will be resumed as a function of the different sonication time of the second step of the preparation method.

Figures 7a, 7b and 7c are characterized by the presence of three well defined zones in which different destabilization phenomena occur. For this reason, in figure 8, relatively to sample 1 as an example, such zones have been identified: the first zone is close to the bottom of the cell, in such a zone sedimentation phenomena occur; the second is representative of the central part of the cell where it is possible to say that flocculation occurs, the third is relative to the top of the cell where clarification occurs. A detailed analysis of the zones has been conducted, taking into account that zone 1 shows a negligible peak and, consequently, it could be analyzed in the same way of zone 3 [11].

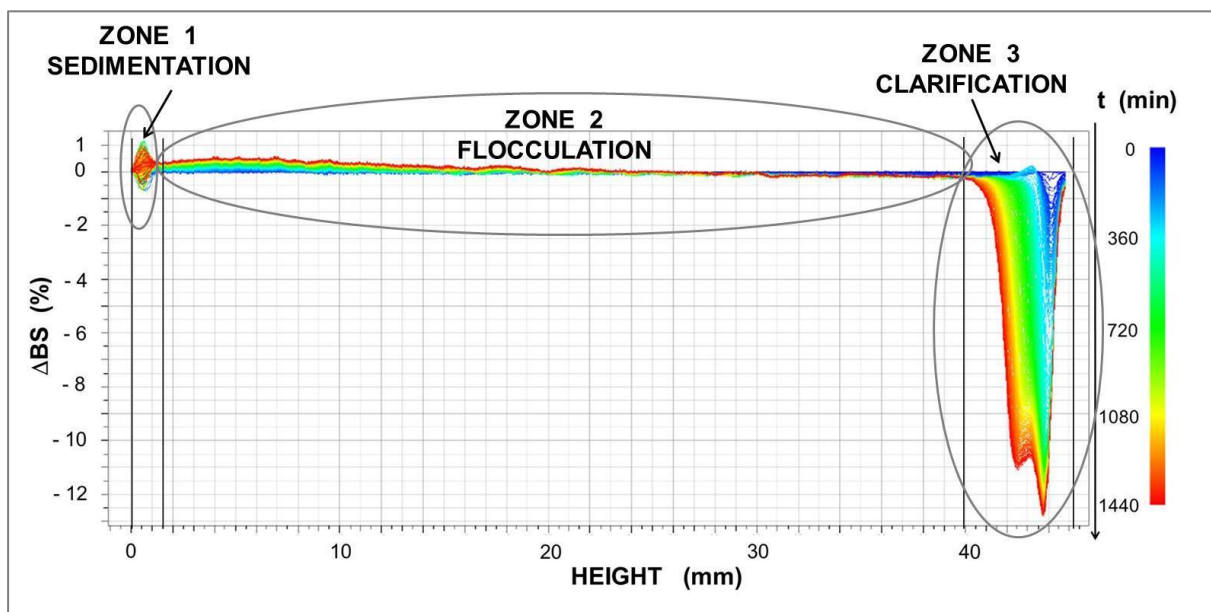


Figure 8. Identification of the three zones where it is possible to distinguish destabilization phenomena: Zone 1: bottom of the cell, sedimentation; Zone 2: center of the cell, flocculation; Zone 3: top of the cell, clarification. Data relative to the sample 1 as example.

3.2.1 Flocculation: analysis of the destabilization phenomena in the zone 2.

Flocculation was analyzed by determining the variation of the particle size during the measurements. For each sample, in the flocculation area, the average variation of backscattering was calculated in terms of average values of the slope of the ΔBS curve as function of time for each cell height in the zone 2.

The analysis of data of sample 1 provides the percentage increase of the particle diameter that corresponds to the slope of the curve that was calculated equal to 0.20% in diameter during the 24 hours of analysis. The analysis has been replicated for the samples 2 and 3 and the percentage increases of the particle diameter have been calculated equal to 0.11% and 0.09%, respectively.

It is evident that the percentage increases of the particle diameter are less significant in zone 2, consequently flocculation phenomena seems to be less relevant, probably also due to the addition of sodium hexametaphosphate as de-flocculant.

3.2.2 Clarification: analysis of the destabilization phenomena in the zone 3.

In the clarification zone, the velocity of clarification was evaluated by the following method [11]: the amplitude of the peak during time at each cell height in the zone 3 has been preliminarily represented

and the average value of the slope of the curve was calculated and from them the velocity of clarification has been estimated. For sample 1 the velocity of clarification is equal to 0.13 mm/hr, for the samples 2 and 3 it is respectively 0.08 and 0.09 mm/hr. Such data have been related to the initial heights of the samples 1, 2 and 3 in the measuring cell, respectively, equal to 45.56 mm, 44.45 mm and 44.72 mm and it was possible to calculate the percentage of clarified phase for each sample equal to 6.48, 1.23 and 0.24% respectively. In order to complete the analysis, the average values of the particles diameter in zone 3 were estimated from the velocity data and the results are reported in Table 5, where the synthesis of all the variation due to destabilization phenomena have been summarised.

Table 5. Synthesis of the effect of destabilization phenomena on the samples depending on the different sonication time of II step.

	ZONE 2	ZONE 3		
	center of the cell, FLOCCULATION	top of the cell, CLARIFICATION		
	percentage increase of the particle diameter %	Velocity of clarification mm/h	Percentage of clarified phase %	Average values of particles diameter μm
Sample 1 <i>sonication time of II step of 10 min</i>	0.20	0.13	6.48	0.47
Sample 2 <i>sonication time of II step of 20 min</i>	0.11	0.08	1.23	0.38
Sample 3 <i>sonication time of II step of 40 min</i>	0.09	0.09	0.24	0.39

It is possible to observe that significant variation could be observed for the sample 1 compared with the other two: higher time of sonication could improve the texture of the materials.

It seems, instead less relevant to improve sonication time until 40 minutes in the II step, when bentonite is added because a very similar behaviour has been observed from samples 2 and 3, as already evidenced also for the thermal analysis.

4. Conclusions

In this paper, thermal analysis and texture characterization of PCMs based on Glauber's salt have been conducted with the aim of demonstrating how relevant the use of multiple light scattering method is, based on Turbiscan instrument, for the evaluation of stability of in-homogeneous materials.

Samples with the same composition were prepared by using sonication as a mixing method with different sonication times with the purpose of verifying the role played by disgregation due to the sonication on the stability of the final product.

Different sonication time seems to affect the disgregation of crystals and consequently the thermal properties and stability during time.

It has been possible to demonstrate that an optimal value of sonication time could be expected in order to improve solidification and melting enthalpy as well as to reduce destabilization effects in terms of flocculation and clarification/sedimentation phenomena.

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